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ARSENIC IN COAL AND COKE.

By Alfred C. Chapman, F.I.C.

(*Read at the Meeting, June* 5, 1901.)

Owing to the necessity of obtaining malting fuel as free as possible from arsenic, the detection and estimation of that element in coal and coke has now become in many laboratories a matter of almost everyday procedure. Up to the present time different methods have been adopted by various chemists, and the somewhat serious discrepancies which have frequently characterized the results are in themselves sufficient evidence that those methods are not altogether beyond criticism. Many analysts have resorted to processes involving the more or less drastic treatment of the fuel with sulphuric acid or nitric acid, or with mixtures of those acids under varying conditions. Others have adopted the method of deflagrating with an alkaline oxidizing mixture, whilst others again have endeavoured to obtain the arsenic by distillation with hydrochloric acid and a reducing agent. For some time I adopted what may be described as an acid extraction method, always, however, with a feeling that it was open to considerable objection. When it became recognised that a considerable proportion of the arsenic in fuel remains in the ash when the fuel is burned, I made a number of determinations of this fixed arsenic, and found that in many cases the results were in excess of those which I had got by the acid process in my endeavour to obtain the total arsenic. It was obvious, therefore, that some other method was required, and after many experiments the process described below was devised. There can be little doubt that the arsenic in coal is present as arsenical pyrites, and that little exists in any other form.

I thought, therefore, that it might be interesting to ascertain how arsenical pyrites itself behaves when strongly heated in the presence of an excess of oxygen. Two samples of pyrites, "A" and "B," having respectively the following composition, were experimented with:

Sample.	Arsenic.	Iron.	Sulphur.
"A" ...	42·32 per cent. ...	33·66 per cent. ...	17·68 per cent.
"B" ...	44·27 ,, ...	34·65 ,, ...	17·34 ,,

Weighed quantities of these were heated to a bright red heat in a porcelain boat placed in a piece of ordinary glass-combustion tubing, the ignition being continued until the weight became constant, and the arsenic left in the residue (apparently as ferric arsenate) was then determined. In the case of "A" this amounted to 8·0 per cent., and in "B" 6·34 per cent. It will be seen, therefore, that a considerable proportion of the arsenic is incapable under these conditions of volatilizing.

When the pyrites was ignited with three times its weight of powdered wood-charcoal, the amount of arsenic remaining in the residue was increased, which may possibly be due to the fact that, owing to the dilution of the pyrites, the heating would necessarily be more gradual, and so increased opportunity for the oxidation of the arsenide would occur.

Before the method described below was arrived at, attempts were made to effect the complete oxidation of the coal by means of boiling concentrated sulphuric acid, as in Kjeldahl's nitrogen process, but so much difficulty was experienced in preventing the extraction of traces of arsenic from the glass that the method was soon given up. It was open, too, to the objection that anthracite coal requires a very considerable time for its oxidation, whilst coke is still more resistant. The method referred to at the commencement of this paper, and which I have now adopted, consists in the gentle ignition of the fuel with a mixture of magnesia and carbonate of soda, and is carried out in the following manner:

A weighed quantity of finely-ground fuel, depending upon the amount of arsenic it contains (usually from 0·5 gramme to 2 grammes will be found to be sufficient), is intimately mixed with about 2 grammes of pure calcined magnesia and ½ gramme of dry sodium carbonate in a deep platinum or, preferably, a silver crucible. This is then supported in a slanting position, and heated by a flame which is just capable of keeping the bottom of the crucible at a dull-red heat, the contents being stirred by means of a platinum wire from time to time. At the end of about one hour the oxidation will be complete, and ½ gramme of pure ammonium nitrate is then added, and the crucible ignited somewhat more strongly for about five minutes. This latter treatment is intended to oxidize any traces of sulphides which might have been formed, and which would, of course, tend subsequently to form insoluble arsenious sulphide, and so to remove it from the sphere of action in the Marsh apparatus. I have no actual proof that this part of the process is necessary, but I think it safer to adopt it. In this way, if care be taken not to heat the contents of the crucible too strongly, a powdery mass will remain, which can be readily removed. This is washed out into a beaker with dilute sulphuric acid, and the solution is then concentrated so as to decompose any nitrate that might have remained after the above ignition. This solution is then submitted to the modified Marsh-Berzelius method, and the mirror or mirrors so obtained are compared with standards. Should the fuel under examination be found to contain an unusually large amount of arsenic, it is obvious that the sulphuric acid solution may be made up to a definite volume and an aliquot portion used in the Marsh apparatus.

The following results will show that this method is accurate with very much larger amounts of arsenic than would ever occur in coal or coke.

In these experiments weighed quantities of arsenical pyrites of known com-

position were mixed with coal in which the amount of arsenic present was entirely negligible in comparison with that which was added, and the mixture treated as described above, save that the arsenic was determined gravimetrically as sulphide :

	Taken.		Found.
Arsenic	0·0050 gramme	...	0·0042 gramme
,, ...	0·0092 ,,	...	0·0096 ,,
,, ...	0·0096 ,,	...	0·0085 ,,
,, ...	0·0063 ,,	...	0·0055 ,,

In the following table numbers are given showing the amounts of arsenic present in six samples of coal, as well as in the coke made on a large scale from these samples, and in the ash, whilst the percentages of ash, of iron, and of calcium, are also given. It may be mentioned that these samples were gas coals, and were selected on account of the appreciable amount of arsenic which they contain :

Sample of Coal.	Arsenic in Coal, Grs. per lb.	Arsenic in Coke, Grs. per lb.	Ash of Coal, per cent.	Arsenic in Ash, Grs. per lb., calculated on the Coal.	Iron in Ash, per cent., calculated on Coal.	Calcium in Ash, per cent., calculated on Coal.	Alkalinity of Ash.
1	1·4	1·7	6·4	0·6	2·27	Trace	Distinct
2	0·5	0·7	16·2	0·3	1·71	Trace	Slight
3	0·7	1·0	6·7	0·3	2·62	None	Neutral
4	0·7	0·6	2·1	0·6	0·73	None	Neutral
5	0·9	1·1	12·5	0·7	1·96	None	Neutral
6	0·8	0·6	12·2	0·3	3·50	1·8	Very distinct

It will be seen that there is no definite relationship between the amount of arsenic present in the coal and that left in the ash on ignition, the proportion being apparently dependent upon the composition of the ash, as well as upon the manner in which the ignition has been performed. There can, I think, be little doubt, as I have stated above, that the arsenic in coal exists almost entirely as a constituent of the pyrites present, and that little exists in any other form. The two following experiments are of interest in this connection. From one of the two samples of coal referred to in the above table the pyritic portions were picked out. An estimation of the arsenic present in this sample showed no less than 1·9 grains per pound, whilst only 0·2 grain per pound was present in the non-pyritic portion. In the second experiment, two samples of New Zealand coal, taken from different parts of the same seam, and giving on analysis the following results, were examined :

	No. 1.		No. 2.
Ash	4·55 per cent.	...	1·60 per cent.
Volatile sulphur ...	2·46 ,,	...	3·22 ,,
Total arsenic ...	0·2 grain per pound	...	0·5 grain per pound
Arsenic in ash ...	0·1 ,, ,,	...	0·18 ,, ,,

Although No. 2 contained much less ash than No. 1, yet in the former there was a distinct quantity of iron, whilst the latter was practically free from that element, and it will be seen that the amount of arsenic in No. 2 was distinctly greater than in No. 1. As examples of the application of this method to higher class fuels than those referred to above, the following numbers obtained with Welsh anthracites of high quality may be recorded :

	No. 1.	No. 2.
Total arsenic ...	0·03 grain per pound	... 0·035 grain per pound
Arsenic in ash ...	0·01 ,, ,,	... 0·020 ,, ,,

That the method described in this paper is capable of yielding approximately accurate results in the analysis of fuel admits of no doubt, and it has the merit of being easily performed, and of involving the use of materials which can without difficulty be obtained perfectly free from arsenic. It is obvious that it might with slight modification be employed in the analysis of other organic materials than coal and coke.

Discussion.

Dr. Morris said that he was inclined to attach more importance than Mr. Chapman to the total arsenic in fuel, for his own experience appeared to indicate that the influence of any arsenic in the ash or dust which accumulated in the kiln on the quantity of arsenic subsequently found in the malt was as great as, or greater than, the influence of the arsenic actually volatilized during kilning. Of course, from the very nature of the contamination in the fuel a serious difficulty existed in connection with sampling. The arsenical pyrites, from which the arsenic was derived, occurred in patches or pockets in the fuel, so that while a sample from one portion of a particular truck-load might be perfectly free from arsenic, a sample from another portion of the same truck-load might be very highly contaminated with arsenic. For that reason he thought it advisable to use for analysis as large a quantity as possible of the fuel, and he thought that the quantity recommended by Mr. Chapman would be far too small to insure anything like a fair average sample of the fuel, while any slight impurity in the reagents used would have a greater percentage effect on the ultimate result than if a larger quantity were worked with. Even working with 50 grammes he had found it difficult to get a really fair sample. The process of analysis he had adopted was practically that which Mr. Chapman had mentioned in the first instance—viz., digestion of the finely-powdered coke or coal with hydrochloric acid to decompose sulphides, and then with fuming nitric acid and sulphuric acid in order to oxidize as far as possible the organic matter, the residue being washed and the extract "Marshed." Even if the whole of the arsenic were not obtained in this way, the figures obtained were comparative, and after all that was what was really wanted. The structure of the kiln had a very great influence with respect to the presence or absence of arsenic in the malt. The whole question was really one which was hardly capable of being dealt with by ordinary methods of analysis, as so many other factors had to be taken into consideration. The question had recently been raised whether arsenic had not always been present to a greater or less extent in beer. In this connection, he might state that in three samples of old beer which he had recently examined (one brewed in 1798, one in 1875, and one in 1886) he had found

respectively approximately $\frac{1}{50}$, $\frac{1}{60}$, and $\frac{1}{75}$ of a grain of arsenic per gallon. These were strong beers, the original gravity ranging from 1090 to 1112. In the case of the two more recent samples he knew that the malt used had been cured with a mixture of anthracite and coke. The first sample was probably brewed before the introduction of coke, but whether the fuel used in that case in drying and curing the malt was coal alone or whether it was a mixture of coal and wood he did not know.

Mr. FAIRLEY said he did not quite agree with Mr. Chapman in trying to apply to the case of coal and coke the inferences drawn from analyses of what might be called pure arsenical pyrites. He had looked up the analyses of a great many samples of pyrites used in the manufacture of sulphuric acid, which might be assumed to be similar in character to the pyrites present in coal, and he had found that very often the percentage of arsenic was approximately 1 per cent. of that of sulphur. Mr. Chapman had referred to the possible influence of different basic constituents in fixing the arsenic. In his own opinion, the substance most likely to fix the arsenic was ferric oxide, which was capable of both reduction and oxidation. In the case of pyrites in which the arsenic formed only a very small proportion of the total pyrites, a larger proportion of the arsenic would be fixed than in cases in which the quantity of arsenic was greater. In regard to coke, he had been in the habit of assuming—though he did not know that he had very good evidence for it—that about one-half of the arsenic he could find was volatile. A good many of Mr. Chapman's figures appeared to agree with this, but it was obviously only a very rough assumption. He was quite certain that Mr. Chapman's method of determining the total arsenic was better than the acid method which had hitherto been used, and which he had always thought could only yield a certain proportion of the total arsenic, and it seemed from the results now obtained that that proportion was not really very much greater than the actual proportion of volatile arsenic. Of course, as Dr. Morris had pointed out, the arsenic carried off in the dust (the quantity of which dust would vary with the strength of the current of gases circulating through the kiln) did not consist merely of volatile arsenic, and might even be arsenic present in some of the ash itself carried off in a finely-divided state. His own recent experiments showed that in kilns of very different construction, made according to what might be called the newest methods and also according to the oldest methods, the dust which collected on the under side of the tiles actually contained as much as 1 per cent. of total arsenic.

Mr. HEHNER desired to ask Mr. Chapman what proportion of carbonate of lime he had used in his attempts to retain the arsenic by this means.

Mr. CHAPMAN said that about equal quantities of arsenical pyrites and of carbonate of lime were used.

Mr. HEHNER said that in a process which had been recently patented the use of lime was proposed for retaining the arsenic. It might be said that as the coal burned the lime became converted into carbonate; but when in practical analytical work the coal was mixed intimately with calcium oxide and heated to a red heat there was very little absorption of carbonic acid by the lime, and it might be taken that the action of actual lime would be different from that of calcium carbonate.

Mr. CHAPMAN said that he had used calcium carbonate because it probably existed in that form in the coal.

Mr. Hehner said it was obvious that some alkaline medium must be used to retain the arsenic, because there were no vessels available by the use of which the complete absence of arsenic could be insured, and some method like that suggested by Mr. Chapman was a necessity. He (Mr. Hehner) was astonished at the very large quantities of volatile arsenic that Mr. Chapman had found in some of the samples of coal referred to. When it was considered that between 5 and 6 pounds of malt were prepared on the average by about 1 pound of coal, it would follow that, altogether disregarding the ash, malt prepared with coal like Mr. Chapman's sample No. 1, which contained 0·8 grain of volatile arsenic per pound, must contain at least 0·16 grain of arsenic per pound.

Mr. Chapman observed that these coals were all gas coals, such as would never be used for malting purposes.

Mr. Hehner, continuing, said that, in spite of what Dr. Morris had said, he thought it important to distinguish between total and volatile arsenic. It was possible, by proper construction of the malt-kilns, to prevent the access to the malt of any considerable proportion of ash, and, as matters at present stood, failure to prevent it by proper construction amounted to culpable negligence. But there was no known method at present of preventing access of volatile arsenic, and it seemed very hard to reject a fuel which might contain, comparatively speaking, a large quantity of total arsenic when practically none of that arsenic might be volatile. He had made a number of experiments, and had found that in most cases the proportion of volatile to total arsenic was far less than one-half. He thought that the present ideas as to the volatility of arsenic would have to be altered. If the question were asked, What happened when an arsenical body was mixed with 90 per cent. of carbon? the reply would be that the arsenic was reduced and volatilized; but this was not actually the case, and some attempt ought seriously to be made to ascertain in what form the arsenic existed. He understood that Mr. Chapman's endeavours to connect it with the iron or any other of the ash constituents had failed. It seemed to him that it must be in a state of organic combination, and that it burned away just as in the case of sulphur, which in coal was undoubtedly partly in a state of organic combination. In endeavouring to retain the arsenic by means of lime, he had found that the lime used was itself not inconsiderably contaminated with arsenic; and on heating some white marble chips in a platinum crucible to red heat and afterwards in a blowpipe-flame, he had found that that again was contaminated with arsenic.

Dr. Morris suggested that, having regard to the temperature below the kiln-tiles and to that at which arsenic trioxide was volatilized, the bulk of the volatile arsenic would probably cease to exist in a state of vapour and be precipitated with the dust, so that really the question of dust was the chief one to be dealt with in regard to contamination.

Mr. Baker thought that, in regard particularly to what Dr. Morris had said and to the way in which malt-kilns were very largely constructed in this country, it would not be of much assistance to return separately the amounts of volatile and of fixed arsenic—at any rate until the present knowledge of the subject was increased. He also had tried to retain the arsenic by means of lime, but found that the lime

contained about $\frac{1}{50}$ of a grain of arsenious acid per pound, a similar quantity being found after the same lime had been ignited to bright red heat for an hour in a muffle, showing that, up to that point at any rate, the arsenic in the lime was fixed. Certainly, so far as malting fuels were concerned, treatment with certain proportions of lime did seem to hold back the arsenic. This he had been able to test on a practical scale with very satisfactory results.

The PRESIDENT (Dr. Voelcker) said that the main point upon which information was wanted seemed to be that referred to by Mr. Hehner—namely, the form in which the arsenic existed, and whether or not the volatile and non-volatile portions were equally harmful. The fact, which had now been so strongly brought out in the case of arsenic, that a considerable proportion remained behind in the ash, was also common to other bodies than arsenic. For instance, if a very highly organic body containing a certain proportion of nitrogen were charred to the state of charcoal, it would seem at first sight that there ought to be no nitrogen left in the charcoal; an appreciable quantity of nitrogen would, however, be found, even though the charring process had been thoroughly carried out. The question seemed somewhat on a par with that of the fertilizing constituents in a soil, which might be partly in an available condition and partly in a non-available or insoluble condition.

Mr. CHAPMAN said that he fully agreed that it was very important to know the proportion of total arsenic as well as that of volatile arsenic, and for this reason he thought that some such process as he had described, which gave both the total and the volatile arsenic, was an advantage. He agreed with Dr. Morris as to the importance of accurate sampling, but he did not agree at all that it was necessary to work on a very much larger quantity than 0·25 or 0·50 gramme. In his own practice he recommended that samples of coal for analysis should consist of about 3 or 4 pounds, representing a much larger and carefully-drawn sample. A thoroughly representative portion was then prepared, from which the small quantity ultimately required for analysis was drawn. It therefore would not matter whether the quantity actually analysed was 0·5 gramme or 50 grammes. In most of these cases, even working upon the small quantities recommended, the mirrors obtained were as dark as could possibly be used for purposes of comparison, 0·25 gramme in some cases yielding a mirror of a depth beyond which it was not possible to go. Even in the case of good anthracite, 1 gramme yielded a mirror as distinct as it was advisable to use for comparison. He must also dissent from Dr. Morris in regard to the application of the "acid method." He had used such a method himself until recently, but regretted having done so after comparing its results with those of the present method, which he knew to be much more accurate. In regard to the question of impurities in the materials, he had found it possible—though only after many experiments and much trouble—to obtain materials which gave what might be termed "perfect blanks" under the conditions of the experiment for half an hour. Any minute trace of impurity that might possibly be present could not have any appreciable influence in cases like those now under consideration, in which the quantities of arsenic were so much greater than those given by average malts, containing, say, from the $\frac{1}{400}$ to the $\frac{1}{200}$ of a grain of arsenic per pound. As a matter of fact, it would be quite impossible to obtain anything like the total quantity of arsenic from such large

quantities as Dr. Morris mentioned. Mr. Fairley had suggested that inferences ought not to be drawn, from experiments with pyrites, in regard to the form in which the arsenic existed in the coal, and he (Mr. Chapman) was sorry to say that, as a matter of fact, it had not been possible to draw any such definite conclusions. He wanted, however, in the first place to see how a substance like pyrites would behave when ignited in the presence of oxygen, and it was interesting to find that about an eighth of the arsenic remained behind in the ash, existing almost certainly as arsenate of iron. He agreed with Mr. Fairley that it was exceedingly probable that the arsenic was fixed chiefly by the iron, and this seemed to be shown by his own experiments with carbonate of lime. In fact, he had never found any appreciable quantity of arsenic in any coal the ash of which contained no iron. He might perhaps repeat that the coals used in these experiments were gas coals and not malting coals. His idea had been that such coals would give larger results, which would possibly have a clearer meaning than the smaller numbers yielded by purer samples.

OIL OF CITRON.

By Herbert E. Burgess.

(*Read at the Meeting, May* 1, 1901.)

Owing to the extensive adulteration of citron oil, considerable doubt attaches to many of the published numbers representing its physical and chemical constants. A sample of the oil of undoubted genuineness having been placed at my disposal, it occurred to me that the results which I have obtained on its examination might possibly be of interest and use to the members of the Society.

There has always been considerable confusion between lemon and citron oil, as in France and Sicily lemon oil is known as "Essence de Citron," while citron is known as "Essence de Cedrat."

Citron oil is stated to be expressed from the rind of *Citrus medica risso;* but, in fact, a smaller variety of the fruit is used, the true citron fruit being used almost exclusively for the production of candied peel.

It is generally known that the majority of citron oil sent to this country is nothing more than a mixture of lemon oil with verbena, and, in some cases, a small quantity of otto of roses is used as a sweetening agent.

The oil referred to above gave on examination the following constants:

Specific gravity, 15° C., 0·8513
Refractive index, $[N]_D$, 20° C., 1·4750
Optical rotation, $[\alpha]_D$, 100 mm. + 80° 13′

On distilling the oil at a pressure of 10 millimetres, the following fractions were obtained:

1. 60–62°	12 per cent.	rotation + 86° 30′
2. 62–64°	80	,,	rotation + 85° 30′
3. 64–85°	5	,,	rotation + 13° 30′

Numbers 1 and 2 were refractionated and distilled several times over clean sodium to remove any oxygenated constituents. The main fraction thus obtained was identified as limonene, boiling at 173° to 174° C. at atmospheric pressure, and having a rotation of +89°. Possibly with this fraction there was a small percentage of the dipentene, which would account for the rotation being somewhat lower than is generally given for limonene, but I was unable to make any further separation.

The third fraction was examined for citral by Tiemann's cyanacetic acid reagent, and I obtained the well-defined citralidene-cyanacetic acid, having a melting-point of 120° to 121° C.

With the mercuric sulphate reagent, as described in the ANALYST, vol. XXV., p. 265, it gave the blood-red coloration peculiar to citral.

A determination of the citral in the original oil gave the following figures:

By sodium bisulphite method (i.) 6·2 per cent.; (ii.) 5·8 per cent.
By hydroxylamine method, 5·7 per cent.

The original oil had a considerable deposit of resinous and crystalline matter, which was filtered off and extracted with chloroform; on standing crystals were formed which were filtered off and dissolved in petroleum spirit to free them from resinous matter. The crystals thus obtained were finally recrystallized from alcohol. Well-formed needle-shaped crystals were obtained, having a melting-point of 145° C., with a marked blue fluorescence in alcoholic solution. The crystals were soluble in alcoholic potash and reprecipitated by acids; quite neutral to litmus; slightly soluble in boiling water, from which they crystallized out on cooling; soluble in strong sulphuric acid. Boiling Fehling's solution was not reduced by them.

Two combustions gave:	I.	II.
Carbon	64·77	64·88
Hydrogen	5·40	5·24
Oxygen	29·83	29·88

Molecular weight determination gave 355, corresponding with a formula: $C_{18}H_{18}O_6$.

I believe this substance to be identical with that observed by Crismer, and more recently by Theulier.

The following table gives my results and those of other workers.

	Specific Gravity.	Rotation.	Boiling-point.	Citral.	Refractive Index.
Sample A ...	0·8513	+80° 13′	60°–100° (10 mm.)	6 %	1·4750
Sample B ...	0·8568	+70° 13′	58°– 90° (10 mm.)	5 %	1·4755
Gildemeister...	0·8710	+67° 8′	177°–220°		
Charabot ...	0·8700	+67° 0′	177°–220°		
Parry... ...	0·86–0·87	+66°–76°			

Sample A represents the genuine oil spoken of above.

Sample B was an oil obtained from another source, and guaranteed to be pure, but on examination it was found to be adulterated with a considerable quantity of lemon oil.

It will be seen at once, by referring to the above table, that the physical con-

stants of a genuine citron oil are widely different from the oils examined by different workers.

I understand that citron oil is only expressed in Sicily to special order.

Discussion.

Dr. Morris asked if the white, needle-shaped crystals referred to were found in the oil itself.

Mr. Burgess said that the oil, as he received it, contained a considerable deposit, which had been filtered off, and from this the crystals were extracted.

Dr. Morris said that some years previously (*Journ. Chem. Soc.*, 1882), in the course of an investigation in connection with the hydrocarbons occurring in resin spirit, he had found in certain samples crystalline needles which behaved with acid similarly to those described by Mr. Burgess, and which he had identified as heptene glycol.

Mr. Chapman said that an important point in connection with this sample was the fact that its rotation was, he believed, some 10° or 12° higher than the highest recorded number for citron oil. The boiling-point of the sample was very much lower than those recorded by Gildemeister and one or two other observers, though it was somewhat doubtful whether any real comparison could be made with these figures, because they represented merely the boiling-points between which the main fraction distilled, whereas those of Mr. Burgess represented the total range from minimum to maximum.

Mr. Burgess said that he had never observed in lemon oil a rotation of over 67°, whereas in this particular oil it was over 80°, or 10° higher than the highest recorded for lemon oil. The specific gravity, of course, was very much lower. The references to the range of boiling-point in the different works on the subject did not indicate at what pressure the boiling-points were determined, but it was probably at atmospheric pressure. The whole range from beginning to end would be taken. Any one particular fraction would have a much smaller range.

THE USE OF PARTIALLY STERILIZED MILK CULTURES IN JUDGING THE PURITY OF WATER.

By H. Droop Richmond, F.I.C.

(*Read at the Meeting, June* 5, 1901.)

Klein has recommended a search for *B. sporogenes enteritidis* as an indication of the purity of water; this is done by inoculating 10 c.c. of sterile milk with 1 c.c. of the water, heating to 80° C. for fifteen to twenty minutes, and incubating anaerobically at 37° C.; a curdling of the casein in dry-looking masses, with enormous evolution of gas, together with a peculiar odour, is characteristic of this organism.

For the past few months I have always made this test, and have also made a second milk-cultivation by inoculating 10 c.c. of sterile milk with 1 c.c. of water, heating to 50° C. for ten minutes, and incubating aerobically at 37° C. I find that it

takes two and a half minutes to heat up 10 c.c. of milk and 1 c.c. of water to the temperature of the bath, and therefore plunge the tubes in cold, and keep them there for twelve and a half, seventeen and a half, or twenty-two and a half minutes, according as I wish to heat ten, fifteen or twenty minutes respectively. There is no difference in the results obtained by heating to 80° C. for fifteen or twenty minutes, and latterly I have always adopted fifteen minutes.

My results with eighty samples of water are given in the tables below. Table I. contains waters which from a careful examination of the source show no signs of pollution; none of these curdled milk heated to 50° C., and only one milk heated to 80° C. Table II. contains waters judged to be free from sewage pollution, but which are more or less exposed, and may be contaminated by decaying vegetable matter and small animals; the bulk of these curdled milks heated to 50° C. in less than three days, and the number of micro-organisms was usually high; chemically there is little to object to in these waters. Table III. contains waters known to be polluted; six out of twenty waters do not curdle milk, and four of these do not give a large number of micro-organisms on cultivation; two, in fact, which are perhaps the worst chemically, and in which the pollution is most plainly established by inspection, are nearly sterile. The other waters in this table all curdle milk heated to 50° within two days.

In Table IV. are classed the doubtful waters; these include waters which change in analysis in six months to an appreciable extent, waters from wells with unprotected sides, waters from springs rising from manured gathering ground, and finally waters about which I have no information.

In two cases, in which pollution was established where chemical examination did not show this, milk heated to 50° was curdled in thirty-six hours, and this was the first indication of pollution.

From a consideration of the results I am of opinion:

1. That any water curdling milk heated to 50° for ten minutes in two days or less is very suspicious.

2. That a water may be considered good if it does not curdle milk heated to 50° for ten minutes, and has a favourable chemical analysis.

3. That some badly polluted waters are bacteriologically good, and these do not curdle milk.

I think that the cultivation of milk heated to 80° has much less value than that heated to 50°, though doubtless the detection of *B. sporogenes enteritidis* would be conclusive; I have never found this organism in the waters I have examined.

Discussion.

Mr. Baker inquired whether the author had been able to differentiate between pathogenic and non-pathogenic organisms by this process. At certain times of the year milk was very apt to be contaminated with *Bacillus subtilis*, which formed spores that would not be destroyed by heating to 50° C.; and although this organism was perfectly harmless, he understood that if it happened to be present in the milk the results of this test would still be taken to indicate that there was ground for suspicion.

TABLE I.—

Source.	Microscopic Examination.	Chlorine.	Free Ammonia.	Albuminoid Ammonia.	N_2O_5	=Nitrogen.
Public supply	good	1·4	0·007	0·0055	trace	trace
Well ...	,,	4·0	0·003	0·006	1·15	0·30
,, ...	,,	1·2	0·002	0·002	1·5	0·39
,, ...	,,	1·4	none	0·0025	1·35	0·35
,, ...	,,	2·0	0·0145	0·0045	none	none
,, ...	,,	2·0	0·0295	0·004	,,	,,
,, ...	,,	1·3	0·001	0·0115	1·45	0·38
,, ...	,,	2·05	none	0·012	1·1	0·29
,, ...	,,	1·4	0·003	0·0045	none	none
,, ...	,,	1·25	none	0·005	1·3	0·36
Public supply	,,	2·3	,,	0·0025	none	none
Well ...	,,	6·4	0·071	0·0075	,,	,,
,, ...	,,	1·8	none	0·007	4·0	1·04
,, ...	,,	1·35	,,	0·004	1·6	0·41
,, ...	,,	1·7	,,	0·005	2·8	0·73
,, ...	,,	1·35	,,	0·0035	1·7	0·44
Spring ...	,,	1·2	,,	0·0095	none	none

TABLE II.—GOOD

Source.	Microscopic Examination.	Chlorine.	Free Ammonia.	Albuminoid Ammonia.	N_2O_5	=Nitrogen.
Spring ...	good	1·3	0·006	0·0175	none	none
,, ...	—	—	—	—	—	—
,, ...	good	1·25	none	0·004	1·1	0·29
,, ...	,,	1·3	,,	0·005	1·2	0·31
,, ...	,,	1·8	0·002	0·0095	none	none
,, ...	,,	1·9	0·0035	0·0145	,,	,,
,, ...	,,	1·55	0·003	0·003	1·9	0·49
,, ...	,,	1·75	none	0·004	2·0	0·52
,, ...	bad	1·7	trace	0·0095	none	none
,, ...	good	0·7	,,	0·012	trace	trace
,, ...	doubtful	1·2	none	0·008	0·4	0·10
,, ...	bad	1·25	0·004	0·007	0·85	0·22
,, ..	doubtful	1·2	0·0065	0·008	1·6	0·41
,, ...	good	2·2	0·0065	0·008	0·6	0·16
Reservoir	,,	2·0	trace	0·0105	0·15	0·04

Mr. CHATTAWAY said that in applying this test he had met with one or two peculiar cases which might perhaps be accounted for in the way suggested by Mr. Baker; but, generally speaking, if coagulation took place, even if the chemical results pointed to the water being fairly good, he thought it worth while to inspect

GOOD WATERS.

Oxygen absorbed in 4 hours.	Gelatin Colonies per c.c.	Agar Colonies per c.c.	Milk heated to 50° C. for 10 minutes.	Milk heated to 80° C. for 15 to 20 minutes.	Remarks.
0·021	10	2	no change	no change	
0·015	50	none	,,	,,	
0·004	10	,,	,,	,,	Abyssinian tube well.
0·006	300	,,	,,	,,	
0·013	none	,,	,,	,,	deep well.
0·027	,,	,,	,,	,,	
0·036	10	,,	,,	,,	
0·036	90	,,	,,	,,	
0·023	none	,,	,,	,,	
0·027	,,	,,	,,	,,	
0·048	,,	,,	,,	,,	
0·038	Pure culture, *B. Aurantiacus.*	,,	,,	,,	deep well.
0·025	120	1	,,	,,	
0·008	40	none	,,	,,	
0·015	2,440	,,	,,	,,	
0·010	none	,,	,,	c. 3 days	
0·067	110	,,	,,	no change	

WATERS WHICH ARE EXPOSED.

Oxygen absorbed in 4 hours.	Gelatin Colonies per c.c.	Agar Colonies per c.c.	Milk heated to 50° C. for 10 minutes.	Milk heated to 80° C. for 15 to 20 minutes.	Remarks.
0·188	1,470	34	c. 36 hrs., gas	no change	gathering ground in wood.
—	very large	9	c. 2 days	,,	
0·017	40	none	no change	,,	travels a little distance exposed.
0·017	2,520	,,	c. 2 days	,,	
0·102	very large	55	,,	c. 2 days	two open tanks.
0·052	,,	141	c. 36 hrs., gas	no change	
0·017	540	2	c. 2 days, gas	,,	loosely-covered tank.
0·008	170	none	no change	,,	
0·096	large	21	c. 24 hrs., gas	c. 2 days	open reservoir.
0·092	48	14	c. 36 hrs., gas	no change	exposed at source.
0·036	40	28	,, ,,	c. 3 days	
0·030	large	1	c. 2½ days	no change	carted three miles in a cart.
0·084	10,000	12	c. 2 days	,,	
0·036	2,220	none	no change	,,	exposed at source.
0·040	320	,,	,,	,,	fed by springs.

the source of the water very carefully. The samples to which he had applied the test were from a district with which he was well acquainted, where the water had been examined very thoroughly, and where, moreover, he rarely analysed a samp without personally seeing the well or other source of supply.

Source.	Microscopic Examination.	Chlorine.	Free Ammonia.	Albuminoid Ammonia.	N_2O_5	=Nitrogen.
Well ...	good	12·95	0·003	0·0095	12·0	3·11
,, ...	fair	0·5	0·004	0·032	trace	trace
,, ...	bad	0·8	0·0165	0·020	none	none
,, ...	good	13·6	none	0·0135	6·7	1·74
,, ...	doubtful	12·9	,,	0·0115	5·4	1·40
,, ...	good	3·3	0·003	0·0105	4·9	1·27
,, ...	bad	13·3	0·002	0·016	27·0	7·00
,, ...	,,	4·9	0·0085	0·020	3·5	0·91
,, ...	doubtful	5·4	none	0·014	4·15	1·08
,, ...	bad	11·1	0·180	0·083	16·7	4·33
,, ...	good	3·55	0·028	0·028	7·5	1·94
,, ...	bad	2·3	none	0·008	1·5	0·39
,, ...	good	6·1	0·057	0·012	15·0	3·89
,, ...	doubtful	11·9	0·0025	0·0115	6·2	1·61
		11·9	0·001	0·0095	9·2	2·39
,, ...	bad	3·2	0·013	0·0085	none	none
,, ...	good	1·55	trace	0·004	1·7	0·44
,, ...	,,	13·8	0·0135	0·0195	6·7	1·74
,, ...	,,	3·05	none	0·006	4·0	1·04
,, ...	doubtful	1·5	0·002	0·004	3·3	0·86
,, ...	good	2·4	0·003	0·0035	7·1	1·84
,, ...	,,	3·55	0·0085	0·0145	7·7	2·0

TABLE IV.—

Source.	Microscopic Examination.	Chlorine.	Free Ammonia.	Albuminoid Ammonia.	N_2O_5	=Nitrogen.
Well ...	good	3·6	0·005	0·016	none	none
,, ...	,,	6·65	0·126	0·010	,,	,,
Public supply	,,	1·3	0·0085	0·011	,,	,,
Well ...	,,	1·1	none	0·0115	0·35	0·09
,, ...	doubtful	1·9	0·0185	0·070	1·5	0·39
,, ...	good	1·8	none	0·0035	3·7	0·96
,, ...	doubtful	1·6	none	0·0045	2·3	0·60
,, ...	good	1·9	0·0045	0·007	none	none
,, ...	bad	2·35	0·0045	0·0115	0·75	0·19
Public supply	good	3·5	0·009	0·006	1·6	0·41
,, ,,	,,	0·65	0·008	0·0025	none	none
Well ...	,,	4·9	0·005	0·0025	,,	,,
,, ...	,,	4·85	0·002	0·0025	,,	,,
,, ...	,,	2·7	0·002	0·004	,,	,,
,, ...	,,	3·15	0·004	0·004	0·85	0·22
Spring ...	,,	1 0	trace	0·004	3·0	0·78
,, ...	doubtful	1·3	none	0·0065	2·4	0·62
Rain-water	fair	0·5	trace	0·0085	none	none
Well ...	good	2·2	0·019	0·006	,,	,,
,, ...	doubtful	2·3	0·0095	0·006	,,	,,
Spring ...	good	1·3	0·0015	0·0045	3·7	0·96
Well ...	,,	2·3	none	0·009	3·0	0·78
,, ...	,,	1·3	0·003	0·0075	1·8	0·47
,, ...	,,	1·6	none	0·0055	2·4	0·62
,, ...	,,	1·4	0·001	0·006	1·3	0·36
,, ...	,,	1·95	0·0065	0·015	2·0	0·52
,, ...	,,	1·8	0·004	0·0075	2·5	0·65

Oxygen absorbed in 4 hours.	Gelatin Colonies per c.c.	Agar Colonies per c.c.	Milk heated to 50° C. for 10 minutes.	Milk heated to 80° C. for 15 to 20 minutes.	Remarks.
0·025	very large	15	c. 36 hrs., gas	c. 3 days	
0·097	„ „	34	„ „	c. 2 days	soft-water well, fed from roof; leaky.
0·107	260	large	c. 2 days	c. 3 days	
0·055	very large	124	c. 36 hrs.	„	half under house.
0·057	1,150	5	c. 2 days, gas	no change	
0·048	620	12	c. 36 hrs., gas	c. 2 days	
0·067	140	67	no change	no change	
0·043	—	—	„	„	
0·046	410	3	„	„	
0·202	170	large	c. 36 hrs.	„	
0·248	20	none	no change	„	
0·040	1,100	*B. coli* present	c. 36 hrs., gas	„	pollution established.
0·046	20	none	no change	„	well between privy, cess-pit, stables and yard.
0·050 0·046	very large	large	c. 36 hrs.	„	under house.
0·112	„ „	„	c. 2 days	c. 3 days	drainage from yard entered through recent opening.
0·017	140	27	c. 2 days, gas	„	slight drainage from yard.
0·107	7,000	17	c. 36 hrs.	no change	
0·021	50	1	no change	„	
0·021	very large	11	c. 36 hrs., gas	„	
0·011	710	large	c. 2 days	„	
0·058	—	none	no change	„	

Doubtful Waters.

Oxygen absorbed in 4 hours.	Gelatin Colonies per c.c.	Agar Colonies per c.c.	Milk heated to 50° C. for 10 minutes.	Milk heated to 80° C. for 15 to 20 minutes.	Remarks.
0·024	130	none	no change	no change	gault waters; probably polluted and purified.
0·038	4,110	6	„	„	
0·134	1,340	1	„	„	
0·063	1,920	10	c. 3 days	„	situated at bottom of a hill; not protected.
0·258	690	3	no change	„	
0·027	60	none	„	„	
0·031	1,450	10	c. 3 days	„	
0·021	240	none	no change	„	sides unprotected from surface-water.
0·065	12,360	4	c. 3 days	„	
0·025	70	none	c. 2 days, gas	„	
0·188	260 B. Fl. Liq.	1	„ „	„	
0·006	large	10	c. 36 hrs.	„	
0·002	340	1	c. 2 days	„	
0·010	20	1	c. 3 days	„	
0·010	40	4	c. 2 days	„	
0·025	360	7	no change	„	gathering-ground manured.
0·023	140	7	„	„	
0·038	90	9	c. 3 days, gas	c. 3 days	
0·009	2,470	1	no change	no change	
0·023	1,440	10	„	„	
0·118	very large	—	c. 2 days, gas	„	gathering-ground manured.
0·059	large	none	no change	„	
0·051	„	„	c. 2 days	„	surface-water finds access.
0·053	very large	large	no change	„	top of well not closed.
0·027	6,000	3	„	„	
0·082	1,780	none	c. 2 days	„	
0·041	very large	„	no change	„	

The President (Dr. Voelcker) remarked that milk was a substance which, as had been pointed out, was from its very nature particularly liable to undergo changes of a kind that would be influenced by other conditions than those which might be due to the water alone. Furthermore, difficulties would probably arise in a test such as this from the fact that a water might contain a certain quantity of dissolved organic matter which would produce results apparently unfavourable, but which might in itself be of perfectly harmless origin.

Mr. Richmond said that the test did not admit of differentiation between pathogenic and non-pathogenic organisms. Of course, if *Bacillus subtilis* were present in the water it would affect the milk. In some cases the action on milk yielded indications which could not be ignored. In one of the instances he had mentioned the existence of the pollution was clearly confirmed by an inspection of the well, though the chemical results were not conclusive.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Determination of Saccharin in Food-Products. H. Défournel. (*Journ. Pharm. Chim.*, 1901, xiii., 512-515.)—The author's method is based upon the facts that saccharin forms a soluble ammoniacal salt on treatment with ammonium hydroxide,

$$C_6H_4\begin{matrix}CO\\SO_2\end{matrix}\!\!>N-NH_4,$$

and that this salt, like all ammonium salts, gives off nitrogen on treatment with an alkaline solution of sodium hypobromite, without decomposition of the saccharin group. The amount of saccharin is then calculated from the volume of gas liberated.

Two hundred and fifty c.c. of the liquid under examination are acidified with sulphuric acid (1 : 10), and shaken in a separating funnel with three successive portions of 50 c.c. of a mixture in equal parts of ether and petroleum spirit. The united extracts are washed with water and evaporated. The residue is treated with ammonium hydroxide, and the excess of the latter evaporated on the water-bath. The ammonium salt is taken up with water and the nitrogen determined in a nitrometer, as in the case of urea.

The volume of nitrogen in tenths of a c.c., divided by the factor 8·9, gives the weight of the saccharin in centigrammes.

The method gives good results when the amount of saccharin is not less than 0·03 gramme, but with smaller quantities it is advisable to concentrate the liquid containing it. C. A. M.

The Examination of Lemon-Juices. E. Spaeth. (*Zeit. für. Untersuch. der Nahr. und Genussmittel*, 1901, 529.)—For the purpose of discriminating between natural lemon-juice and that artificially prepared from citric or tartaric acid, determinations of the ash and the alkalinity of the ash are the most useful. Natural lemon-juice contains about 0·4 per cent. of ash, using about 6 c.c. per cent. of normal acid for neutralization, whilst citric acid solutions have no appreciable ash, with, of course, no alkalinity. An estimation of added tartaric acid, if any, is of some value, and may be carried out in the usual way, after first precipitating the organic acids as their lead salts in alcoholic solution, and decomposing these salts with sulphuretted hydrogen. W. P. S.

Sitogen: a Vegetable "Meat Extract." A. Beythien. (*Zeit. für. Untersuch. der Nahr. und Genussmittel*, 1901, 446.)—In this paper is given an analysis of sitogen—an extract prepared from yeast. Coagulable albumen is practically absent. A quantitative analysis of the extract gave the following results:

Water	29·02 per cent.
Insoluble in water	0·38 ,,
Mineral matters	21·25 ,,
Phosphoric acid	5·56 ,,
Chlorine	5·19 ,,
Total nitrogen	7·01 ,,
Total acidity (as lactic acid)	3·38 ,,
Volatile acidity (as acetic acid)	0·16 ,,
Ether extract	0·74 ,,

The total nitrogen contained 0·43 per cent. present as ammonia (0·52 per cent.), 1·38 per cent. as albumoses (8·63 per cent.), and 5·15 per cent. as peptones and bases (32·19 per cent.). The mineral matter was composed of sodium chloride, 40·33 per cent.; phosphoric acid, 26·17 per cent.; sodium oxide, 15·12 per cent.; potassium oxide, 11·81 per cent., etc. The sodium chloride is added to the extract, and does not come from the yeast. W. P. S.

Lloyd's Test for Morphine. J. L. Mayer. (*Amer. Journ. Pharm.*, 1901, lxxiii., 353-355.)—In this test a small amount of hydrastine is mixed with the substance and a few drops of strong sulphuric acid added, a violet-blue coloration being obtained after five minutes in the presence of morphine.

Conversely, hydrastine can be detected by the addition of morphine and sulphuric acid.

The author has applied this test to other alkaloids, using approximately 1 part of hydrastine with 8 parts of the given alkaloid, and has noted the final coloration after stirring the mixture for five minutes.

The following colorations were thus obtained: Aconitine, brown; atropine, pinkish; berberine, greenish-brown; brucine, light brown; caffeine, dirty white; cinchonine, dirty yellow; cinchonidine, dirty white; cocaine, unaffected; codeine, pinkish; digitaline, mahogany; heroine, violet to purple; homatropine, pale yellow; hyoscyamine, dirty white; morphine, violet-blue; pilocarpine, light brown; quinidine,

light green; quinine, greenish-yellow; sparteine, greenish-yellow; strychnine, dirty white; veratrine, royal purple.

From this it appears that only heroine, morphine, and veratrine give a violet-blue colour in this test.

Of these, only veratrine gives a cherry-red colour with strong sulphuric acid; whilst morphine can be differentiated from heroine by means of nitric acid, the former giving an orange-red colour and the latter a yellow one. C. A. M.

Chemistry of the Jaborandi Alkaloids. H. A. D. Jowett. (*Brit. Med. Journ.*, October 13, 1900.)—The author has investigated the chemical properties of the four members of this class of alkaloids, the following being the results obtained:

Pilocarpine, $C_{11}H_{16}O_2N_2$, was formerly obtained from the leaves of true jaborandi (*Pilocarpus jaborandi*), but owing to the scarcity of these leaves the chief sources are the leaves of *P. pennatifolius* and Maranham jaborandi (*P. microphyllus*). These leaves may contain as much as 0·5 per cent. of the alkaloid. It is a thick syrup, yielding crystalline salts. The nitrate is fairly soluble in water, sparingly in strong alcohol. The hydrochloride is very soluble in water and moderately so in alcohol. The commercial nitrate is fairly pure, but contains a varying amount of isopilocarpine nitrate. The author suggests that, as standards of purity, the melting-point of the nitrate should be from 173° to 178°, and its specific rotation +80° to +83°. As additional tests, the behaviour of a strong solution with excess of ammonia, and the formation of a crystalline picrate melting sharply at 147°, are suggested.

Isopilocarpine occurs with pilocarpine, and may be formed by heating the latter with water in a sealed tube for four hours at 180°. In its chemical properties it closely resembles pilocarpine, and the two cannot be easily separated by fractional precipitation. The greater amount of isopilocarpine found in pilocarpine is formed during the process of manufacture. In alkaline solution optically active pilocarpine is converted into the optically inactive isopilocarpine. In neutral solution isopilocarpine is optically active.

Pilocarpidine, $C_{10}H_{14}O_2N_2$, is found only in the leaves of true jaborandi. The nitrate is soluble in twice its own weight of water, and has a melting-point of 137°.

Jaborine.—No alkaloid answering to the description of jaborine is to be found in either true or Maranham jaborandi, and the jaborine of commerce is impure pilocarpine.

The Constitution of Pilocarpine.—Isopilocarpine, which, as is seen above, can readily be obtained by heating pilocarpine, acts not only as an ammonia base, but as a lactone, forms salts with hydrates, and can even be titrated with hot alkali. On oxidation it yields ammonia, methylamine, acetic acid, and an acid of the formula $C_7H_{10}O_4$, which is probably the lactone of hydroxy-isobutyl-malonic acid. Iso-butyric acid is formed by the fusion of pilocarpine with caustic potash. Methylamine alone is formed by the action of potash on methyl-isopilocarpine. There are thus indications of the following groups in isopilocarpine, $(CH_3)_2 : CH.CH.CH(CO_2)C : NH : NCH_3$, leaving three atoms of carbon and of hydrogen to be accounted for. W. P. S.

The Aniseed Oils and Anethol. G. F. Pancoast and L. F. Kebler. (*Amer. Journ. Pharm.*, 1901, lxxiii., 356-359.)—The seed from various sources yields from 1·5 to 6 per cent. of oil. Occasionally stems, chaff and leaves are added to the seed before distillation. The chaff contains about 0·5 per cent. of oil.

It is asserted that aniseed oil can only be distinguished from star anise (*Illicium verum*) by the odour and taste; and according to the authors none of the chemical tests are satisfactory.

Star anise oil is largely manufactured by the Chinese, and is exported in tin cans holding from 42 to 46 lb. Owing to its cheapness it has to a large extent replaced the genuine oil.

The physical properties of star anise oil resemble those of aniseed oil, but its composition is somewhat more complex.

The following results were obtained by the authors in the examination of various aniseed oils sold as pure.

Source.	Specific Gravity.	Optical Rotation.	Congealing Point.	Solubility in Equal Volume of Absolute Alcohol.
1. Russian ...	0·9838 at 17° C.	+3° 50′	+15° C.	Soluble.
2. ,, ...	0·9893 at 20° C.	−4° 59′	18° C.	,,
3. Tonquin ...	0·9834 at 17° C.	−1° 30′	17° C.	,,
4. Star anise ...	0·9648 ,,	−1° 27′	15° C.	,,
5. ,, ,, ...	0·9870 ,,	+0° 58′	16° C.	,,
6. ,, ,, ...	0·9822 ,,	−1° 53′	15·5° C.	,,
7. ,, ,, ...	0·9821 ,,	−1° 31′	14·5° C.	,,
8. ,, ,, ...	0·9832 ,,	−1° 44′	14° C.	,,

As regards these results, the authors considered that oils No. 1 and 2 had been tampered with, the first probably by the addition of oil of fennel. No. 8 was also abnormal, and possibly contained star anise leaf oil, which has a specific gravity of 0·9878 at 15° C., and an optical rotation of +1°. The amount of anethol in star anise leaf oil is small, and its congealing point correspondingly low.

Pure oils with a low congealing point are sometimes met with. These are obtained from a mixture of natural and artificially-ripened seed, and are known as "Flower Oils." In the author's opinion they are inferior to oil made entirely from ripe seed.

Anethol.—According to the German Pharmacopœia anethol is a colourless, highly refractive liquid, with an odour of pure anise. Its specific gravity at 25° C. is 0·984 to 0·986. It melts at +20° to 21° C., boils at 232° to 234° C., and forms a clear solution with two parts of alcohol.

Several samples examined by the authors gave the following results:

	Specific Gravity.	Optical Rotation.	Congealing Point.	Boiling Point.
A	0·9895 at 20° C.	inactive	+17° C.	210° to 235° C.
B	0·9896 at 20° C.	−1° 30′	+20° C.	220° to 235° C.
C	1·0525 at 15° C.	−2° 18′	—	228° to 245° C.
D	0·9870 at 20° C.	+5° 22′	+20° C.	229° to 236° C.
E	0·9847 at 20° C.	inactive	+19° C.	210° to 235° C.

All dissolved in an equal volume of alcohol. Of these, the authors regarded A, B, and E as of fair quality, although anethol is usually considered to be optically inactive. C and D were labelled "liquid anethol," by which is indicated a redistilled oil of anise prepared from ordinary aniseed oil. C was an old sample, and therefore had possibly altered in composition. D was considered to be "anethol" derived from oil of fennel. C. A. M.

Saffron Adulteration and Saffron Essence. **W. Fresenius and L. Grünhut.** (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1900, iii., 810.)—Two samples of adulterated saffron were examined by the authors with the following results:

	I.	Per Cent.
Adulterants	Magnesium sulphate ($MgSO_4$, $7H_2O$)	25·50
	Borax ($Na_2B_4O_7$, $10H_2O$)	8·23
	Neutral sodium borate ($Na_2B_2O_4$, $8H_2O$)	17·49
	Hygroscopic moisture	2·05
	Other constituents (difference)	46·73
		100·00

	II.	Per Cent.
Adulterants	Potassium nitrate	12·94
	Neutral potassium borate ($K_2B_2O_4$, $3H_2O$)	20·86
	Neutral sodium borate ($Na_2B_2O_4$, $4H_2O$)	6·41
	Sodium hydroxide	3·21
	Hygroscopic moisture	8·43
	Other constituents (difference)	48·15
		100·00

Caustic soda appears to have been added in both cases, in sufficient quantity in I. to convert part of the borax into neutral borate, and in II. to convert the whole into neutral borate and leave an excess, the object being apparently to increase the solubility of the added borax.

It is worthy of note that salts should have been selected which contain water of crystallization, which is only partially expelled on drying at 100° C.; thus, magnesium sulphate loses at 100° C. only 5 of its 7 molecules of water, and borax only 8 of its 10 molecules. A partial analysis, showing water lost at 100° C., ash and saffron by difference, would consequently be incorrect, and more particularly so since the determination of water lost at 100° C. would itself be inaccurate, owing to the absorption of carbonic acid by neutral sodium borate on drying.

The authors further examined a sample of so-called saffron essence, which has been placed upon the market. This article was found to have the following composition: Water, 46·57; borax crystals, 16·87; potassium hydroxide, 8·94; potassium nitrate, 10·03; saffron, 0·40; cane-sugar, 9·91; dextrose, 1·65; dextrin, 5·63. The authors suggest that it is simply the liquid which has been used for sophisticating saffron, as explained above, and that the saffron on being immersed in it has imparted to it some of its colouring and extractive matter. H. H. B. S.

ORGANIC ANALYSIS.

Identification of "Renatured" Spirit. P. N. Raikow and P. Schtarbanow. (*Chem. Zeit.*, 1901, xxv., 434.)—This is a process for the detection and approximate estimation of methyl alcohol in renatured spirit, and depends (*a*) on the partial oxidation of the liquid to the respective aldehydes by means of a glowing platinum wire, (*b*) on the easy organoleptic detection of formaldehyde even in presence of a large excess of acetaldehyde by the mucous membranes of the nose and eye. The apparatus employed—it is important to observe the dimensions, etc., specified—consists of a conical flask of such size and charged with such a volume of liquid (say, a 100 c.c. flask and 10 to 20 c.c. of spirit) that the space not occupied by the alcohol shall be equal to 70 or 100 c.c., and shall be about 10 centimetres in height. In the neck of the flask is hung a spiral of platinum made of wire 0·3 millimetre in diameter. The alcohol is dehydrated by means of potassium hydroxide, and a small quantity is also put into the liquid to take up the water formed during the oxidation, but it is not necessary to remove the aqueous layer. The alcohol may be gently warmed, care being taken that its vapour do not expel all the air from the flask. The spiral is heated to redness in a flame, then dropped into position in the neck of the flask; it continues to glow steadily, and copious vapours are given off. The nose is held a few centimetres above the opening of the flask and the fumes are inhaled. From experiments on pure ethyl and methyl alcohol it appears that the vapours from the former can be breathed for minutes at a time; from methyl alcohol the vapour cannot be inspired twice. From 10 per cent. of methyl alcohol (90 per cent. of ethyl) inspiration is only possible for two or three seconds; from 2 per cent. thereof the peculiar odour of formaldehyde can be perceived in about fifteen seconds; the limit of delicacy by the nose being at about 1 per cent. The eye is somewhat more sensitive, but indications of 1 per cent. of methyl alcohol are doubtful. By distilling 100 c.c. of 99 volumes of ethyl mixed with 1 volume of methyl alcohol in an ordinary fractionating flask and collecting the first 10 c.c. which passes over, the odour is about equal to that of a liquid containing 2·5 per cent. of methyl alcohol. Similar experiments on German duty-free spirit from which the pyridine bases had been precipitated with phosphoric acid and the liquid concentrated (or not) by distillation were equally satisfactory.

The method may be rendered somewhat more elegant by placing the flask under an apparatus by the aid of which the evolved vapours can be aspirated through two flasks charged with water. The aldehydes condense, but the acetaldehyde can be easily driven off by heat or by continued aspiration, whereas the formaldehyde remains in the liquid, and will be found in the first condensing vessel. Fifty c.c. of pure ethyl alcohol may be oxidized in this apparatus (in nine hours), yielding liquids which reduce ammoniacal silver nitrate before or after evaporation to a small bulk, but which are free from the odour of formaldehyde. In presence of 1 per cent. of methyl alcohol the liquid in the first vessel will cause the eye to weep. It will be apparent that this test depends subjectively on the absolute quantity of formaldehyde in the liquid smelt, and that in the first form its chemical delicacy depends on the relative amount produced and volatilized in a given time, so that there is a distinct limit to

its usefulness. In the second modification any quantity of alcohol can be decomposed by prolonging the time of treatment, and as the whole of the methyl alcohol will be recovered as formaldehyde, it serves to detect a smaller amount of the lower alcohol.

Preliminary experiments with ether and acetone show that in pure acetone the wire glows more brightly than in alcohol. The vapours have only a faint odour, which, like that of acetaldehyde, is felt at the anterior part of the nose, formaldehyde affecting the posterior portion. The vapours from acetone can be breathed for several minutes. One per cent. of methyl alcohol in acetone, accordingly, can be safely detected. With ether the spiral has to be supported just above the neck of the flask, when it will continue to glow for a few seconds. The vapours produced from ether are extremely irritating, but quite different from formaldehyde in odour, so that 2 per cent. of ether in acetone can be recognised. Two per cent. of ether in alcohol give a doubtful indication. The nature of the vapours evolved from acetone and ether is not yet known. F. H. L.

Existence of Salicylic Acid in Wine. H. Mastbaum. (*Chem. Zeit.*, 1901, xxv., 465.)—In 1890 Medicus first called attention to the fact that certain undoubtedly genuine wines gave on extraction a substance yielding the violet colour with ferric chloride, which is supposed to be characteristic of salicylic acid. Sufficient of the material could not be obtained for the identification of its true nature; for, in fact, the test usually fails unless 100 or 200 c.c., or even a larger volume of wine is taken for examination. Hence, Medicus recommended that only 50 c.c. of a wine should be employed in seeking for added salicylic acid, and his proposal has been officially adopted in Germany. This method (extraction with equal volumes of ether and petroleum spirit) will indicate as little added salicylic acid as 0·5 gramme per hectolitre, *i.e.*, 0·25 milligramme per 50 c.c.

Mastbaum has attempted to discover whether Medicus' substance is actually salicylic acid, or whether, as Simon has suggested, it may be isopyrotritaric acid (*Compt. Rend.*, 1900, cxxxi., 618). A Torres Vedras wine was taken which was represented by the Tony-Garcin test to contain 0·9 milligramme of "salicylic acid" per litre. Five litres were concentrated to 500 c.c. and extracted with ether and petroleum spirit. Water was added, the volatile solvents removed, and the aqueous liquid treated with chloroform. From the latter a yellowish crystalline mass was obtained, which was dissolved in hot water, decolorized with animal charcoal, and filtered. The filtrate was extracted once with chloroform, afterwards with ether. These solutions, and especially the ether, yielded white needles which melted sharply at 155° C., sublimed above that temperature, and when they were dissolved in water gave the ferric chloride reaction strongly, also the Millon-Lintner test (ANALYST, 1900, xxv., 274), and Jorissen's reaction with sodium nitrite, etc. The yield was not large enough to determine the molecular weight and other constants, but there seems no doubt that the body is true salicylic acid, more particularly since some wines give the violet test more strongly after heating on the water-bath with 1 per cent. sulphuric acid than they do originally. F. H. L.

A New Test for Sugar. T. Sollmann. (*Centralbl. Physiol.*, 1901, xv., 35; through *Chem. Zeit. Rep.*, 1901, 157.)—The reagent is a variant of Fehling's solution, prepared by mixing 50 c.c. of 10 per cent. sodium carbonate and 50 c.c. of 5 per cent. Rochelle salt with 10 c.c. of 1 per cent. cobalt nitrate or nickel sulphate. With nickel the reagent is apple-green; heated to boiling, and a little dextrose solution added, it gives a pale to canary-yellow colour, according to the proportion of sugar, the liquid remaining quite clear. The cobalt reagent is at first almost colourless, but in half an hour becomes bluish-green. Heated to the boil (alone), it turns sky-blue, becoming green again on cooling; soon a precipitate forms, and its delicacy falls off. If dextrose is added to the boiling cobalt reagent, the colour changes through emerald-green to yellow-green and reddish-brown. Traces of sugar produce only a pale green; large quantities yield a brown like iodine solution. These tints are permanent for days. The colours only develop at the boiling-point. The tests are one and a half to two times as delicate as those with copper.

The reactions succeed with dextrose, invert sugar, galactose, and the sugars produced by boiling gums with acids, also with aldehydes and various gums, the latter of which fail or give only a green colour with Fehling's solution. The reactions do not appear with saccharose, mannite, glycogen, dextrin, starch; aliphatic bodies such as acetone, alcohols, acids, and fats; aromatic substances, the xanthine group, creatinine, urea, alkaloids, glucosides, amido-acids, and inorganic reducing and oxidizing agents. With albuminoids colour changes appear, which, when typical, are different from the sugar tints; sometimes, however, they resemble them. F. H. L.

The Baudoin, Tambon, and Soltsien Reactions for Sesamé Oil. Utz. (*Chem. Zeit.*, 1901, xxv., 412.)—This article consists largely of a historical review of the Baudoin reaction, a copious list of references to the literature of that test being given. The author has examined Tambon's test (Analyst, this vol., p. 105), using a 4 per cent. solution of dextrose in 1·19 hydrochloric acid. This reagent develops no colour of itself, at least in the cold, and it keeps well. On heating (alone) it gradually gives an orange colour, which, however, cannot be mistaken for the sesamé tint. With pure sesamé oil no colour appears on shaking in the cold for half a minute; on heating, the pink to red colour is produced, and attains its maximum in three to five minutes. The limit of delicacy appears to be at 2 per cent. of sesamé in an oil. Old or rancid sesamé gives a reaction: if pure, the colour is red-brown; mixed with other oils, pale brown. African sesamé yields the strongest colour, then Levantine oil, and finally Indian; this is the same phenomenon as obtains with the furfural test. Spectroscopically, the red Tambon mixture exhibits certain differences from the products of other tests; but the reaction cannot be recommended for general purposes.

Utz calls attention to the Soltsien test with stannous chloride. Properly carried out, it is the simplest, most certain, and most delicate of all; and it is particularly valuable when confirmatory evidence of the presence of sesamé is required.

F. H. L.

Apparatus for Determining the Melting-point of Gelatin-Jelly, Fats, etc. N. Chercheffsky. (*Chem. Zeit.*, 1901, xxv., 413.)—This apparatus consists of a 250 c.c. beaker filled with pale petroleum oil, and having a glass rod laid across its top. One end of a Z-shaped brass wire is coiled round the rod, the other depends into the liquid with its horizontal portion level with a thermometer bulb. On this end of the wire are skewered little cubes of the solidified gelatin solution, about 5 millimetres long, the jellies being prepared from equal weights of gelatin and water. For greater accuracy the beaker may be supported on cork blocks inside a water-jacket. By filling the inner vessel with water instead of oil, the melting-points of fats, waxes, and the like may be similarly ascertained. F. H. L.

A New Method for the Estimation of Soluble Nitrocellulose in Gun-cotton and Smokeless Powder. K. B. Quinan. (*Journ. Amer. Chem. Soc.*, xxiii., 258.)—The two methods now in use are the "aliquot" method, and the "residual" method, used by the United States Government. In the first, about 1 gramme of the dried and finely-divided sample is treated in a closed vessel with 250 c.c. of a mixture of two parts of ether and one of alcohol. After all the soluble matter has been brought into solution by shaking, an aliquot part is transferred into a small tared flask, evaporated at 65·5° C., dried at 100°, and weighed. This method gives good results, but requires very careful working.

In the second method, 1·5 grammes of the sample are agitated in a covered vessel for at least two hours, with 250 c.c. of the same mixture of alcohol and ether; the residue is allowed to settle, the solution filtered through a tared asbestos filter, the residue again treated with 200 c.c. of ether-alcohol, the solution filtered, and the asbestos filter and residue, after thorough washing, are dried first at 40° and then at 100°, and weighed. The loss represents the soluble matter. By this method four or five days are required for an analysis, rendering it practically useless for manufacturers.

In the author's "centrifugal" method, 1 gramme of the sample is placed in a small cylindrical aluminium vessel; 50 c.c. of alcohol are added, and the sample is thoroughly stirred. After the addition of 100 c.c. of ether the whole is again stirred for several minutes, the vessel covered with a loosely-fitting cap, and placed in one of two cups mounted on the end of a horizontal shaft, which can be rotated at a speed of about 2,000 revolutions per minute by means of a vertical driving-shaft. The power necessary for this is about ½ h.-p. In the other cup of the horizontal arm a second sample may be placed, so that two analyses can be carried out at the same time. After the sample has been agitated for about ten or twelve minutes the aluminium vessel is removed from the cup, when it will be found that the insoluble matter has settled completely at the bottom of the vessel. Almost the whole of the clear liquid can now be drawn off by means of a vacuum pipette provided with two stopcocks in its lower and upper stems; the pipette is exhausted before use, and both its stopcocks are closed; the lower stem is then immersed in the solution, and the lower stopcock opened, when the liquid will enter the pipette. To the residual 10 or 15 c.c. of solution in the aluminium vessel 50 to 75 c.c. of fresh ether-alcohol are

added, the whole is stirred and again revolved in the centrifugal machine, and the liquid removed as before. This process is repeated until all the soluble matter has been removed, which can usually be done in seven or eight washings, though samples containing much insoluble matter may require twelve or more washings. The insoluble matter is then transferred to a Gooch crucible and dried at 100°, or dried directly in the aluminium vessel, and weighed.

Exclusive of the time required for drying, an analysis can be completed by this method in from one to two hours, as each successive extraction requires a shorter time in the centrifugal machine, since the viscosity of the solution decreases each time. The results obtained agree well with those given by the " aliquot " method.

A. G. L.

INORGANIC ANALYSIS.

The Measurement of Gold and Silver Buttons in Quantitative Blowpipe Assays. Joseph W. Richards. (*Journ. Amer. Chem. Soc.*, xxiii., 203.)—Beside actual inaccuracies in construction, the Plattner ivory scale possesses several defects, chief amongst which is the difficulty of getting the round buttons to the right spot on the flat surface of the scale. To remedy this, the author has contrived a scale consisting of two perfectly straight aluminium edges lying on a flat surface, touching each other at one end, and held apart at the other by a set-screw, so that the point 100 on the scale indicates a separation of 1 millimetre. In using the scale, the button is put into the groove, the scale inclined, and tapped until the button wedges itself in. Since the buttons are not perfectly spherical, the highest of several readings should be taken as the true horizontal diameter. In order to render them more uniform in shape, the cupelled buttons are remelted on charcoal before measuring, as recommended by V. Goldschmidt. One of the metal strips is pressed against the set-screw by a small spring which allows it to be pushed back, thus facilitating the removal of buttons and cleaning of the scale.

If the buttons consist only of gold or of silver, their weight may be obtained directly from a table given in the paper. If both gold and silver are present, the determination of silver is greatly simplified by the use of the case of standard alloys designed by Goldschmidt. In the first place the button is measured, and its volume obtained from the table. It is then flattened out and compared with the standard alloys, under a lens, by diffused daylight. The percentage of silver is thus determined approximately. The specific gravity of the alloy is then obtained from a table also given in the paper, and, on multiplying the volume by this, the weight of the original button is obtained, from which the weight of gold and silver can be found.

This method cannot be applied to buttons containing more than 56 per cent. of silver, since all these alloys are silver-white. In this case the best plan is to measure the button and obtain its volume from the table, then replace it on charcoal, and heat intensely in the oxidizing flame, when the silver will slowly volatilize. As soon as the button has a pronounced yellow colour, which will be the case after one to five minutes, it is again measured, flattened out, and compared with the standard alloys. The weight of gold present may then be found as before, and on taking the volume

corresponding to this weight from the volume of original button, the volume of silver present is obtained, from which its weight may be calculated. Another method consists in melting the original button with a button of pure gold of about the same (known) diameter. The button obtained is measured, compared with the standard buttons, and the weights of silver and gold obtained as before. The weight of gold added is then subtracted from the total weight of gold, and the remainder gives the weight of gold in the assay. All these methods are based on the fact, which has been experimentally verified, that gold and silver neither expand nor contract on alloying with each other. A. G. L.

Estimation of Zinc with Iodine Solution. **P. Knaps.** (*Chem. Zeit.*, 1901, xxv., 539.)—In acetic acid solution, zinc sulphide reacts quantitatively with iodine to form zinc iodide and sulphur. But in solutions containing more than 0·05 gramme per 200 c.c. of water, part of the sulphide and the liberated sulphur cohere, and prevent the completion of the decomposition. This, however, can be avoided by producing within the liquid some indifferent precipitate such as barium sulphate, which iodine does not attack. The original zinc solution is treated with 10 c.c. (for every 0·05 gramme of zinc) of a solution of 150 grammes of crystallized barium chloride per litre, and an equal volume of a solution of 200 grammes of crystalline sodium sulphate per litre is added. The zinc is then thrown down with sulphuretted hydrogen, the excess of the gas is driven off by sharp boiling till a lead paper is no longer discoloured by the vapours, the liquid is cooled, excess of standard iodine is run in, shaken for a minute or two, and the whole titrated, as it is, with thiosulphate. Reasonable amounts of manganese salts do not interfere; nitrates must be absent. The results quoted appear satisfactory, even in presence of manganese. F. H. L.

Detection of Indium. **P. Kley.** (*Chem. Zeit.*, 1901, xxv., 563.)—The best substance for the detection of indium is the double chloride of that metal with rubidium, which is produced by adding a crystal of rubidium chloride to a solution of indium hydroxide in strong hydrochloric acid. The crystals appear as colourless octahedra which polarize light and belong to the rhombic system; they measure 10 to 70 μ. The limit of delicacy of the reaction is at 0·00024 milligramme of indium. By employing cæsium chloride instead of rubidium chloride, the test is ten times more delicate, but the crystals are so small as to be difficult of recognition. Indium can be separated from other metals by treatment with ammonia; the hydroxide adheres to the microscope object glass, and can be washed with ammonia and water till the rest of the zinc, cadmium, etc., is removed. Neither aluminium nor zinc affects the process. If 50 per cent. of zinc is mixed with the indium, the precipitation with ammonia is not necessary; but if the indium is accompanied by large amounts of trivalent metals, the liquid should be treated with ammonia, the indium hydroxide dissolved in ammonium carbonate, and then thrown down with oxalic acid. When, however, the quantity of indium is extremely minute, the rubidium chloride reaction is better; but all ammonia, and as much ammonium chloride as possible, should be removed by heating, or the crystals will be very small.

Ammonium chloride yields a double salt with indium chloride as rubidium and cæsium do. The ammonium compound is readily soluble in hydrochloric acid, like the potassium compound.

Huysse has recently recommended a micro-chemical test for indium based on the formation of cæsium-indium alum. Even in strong solutions indium sulphate with a little sulphuric acid yields the above-mentioned double chloride when cæsium chloride is added. The reaction succeeds under similar conditions if cæsium nitrate is adopted; but it is useless in presence of aluminium. Kley has not been able to obtain the reaction with ammonium mercuric thiocyanate, also mentioned by Huysse, even in highly concentrated solutions. Hitherto this reaction has only been known to occur with divalent metals. Huysse's description reads like that of the corresponding zinc salt; and as indium is recovered from zinc-dust, it seems possible that zinc is the cause of the reaction. Huysse's oxalate test only works in ammoniacal solution, and is valueless when but little indium is present. F. H. L.

Volumetric Estimation of Chromium in Chrome Mordants. R. Hartmann. (*Chem. Zeit.*, 1901, xxv., 564.)—About 1 gramme of the chrome acetate liquor (of 21° Beaumé) is diluted with 10 or 15 c.c. of water, and in a 1-litre porcelain basin is treated with strong sodium hydroxide till the precipitate redissolves. At the temperature of the water-bath sodium peroxide is next added until the liquid is pure yellow. It is then evaporated to dryness in order to destroy traces of peroxides, dissolved in 500 c.c. of water, made acid with dilute sulphuric acid, and mixed with 50 c.c. of decinormal ferrous ammonium sulphate. Finally the solution is diluted to 1 litre, and titrated with permanganate till the green colour changes to a dirty violet. At the dilution prescribed, the end-point is quite sharp. Acetate prepared from bichromate and molasses, starch, etc., must first be freed from reducing matter by means of nitric acid or hydrochloric acid and chlorate, then converted into sulphate as above. The organic matter in commercial acetate reacts with permanganate, and causes the amount of chromium oxide to appear a little too low, but the error is only 0·02 to 0·09 per cent., and for commercial work may be ignored. The results quoted are satisfactory. F. H. L.

On the Quantitative Precipitation of Metals by Organic Bases. W. Herz. (*Zeit. anorg. Chem.*, xxvii., 310.)—In pursuing his experiments on the precipitation of metals by organic bases possessing high dissociation constants (*Zeit. anorg. Chem.*, xxvi., 90 and 347), the author found that copper could be quantitatively precipitated by adding a solution of guanidine or piperidine to the solution of the copper salt, slightly warming for a short time, filtering the precipitate, and igniting as usual to CuO. Magnesium and zinc were also completely precipitated from solutions of their sulphates by the addition of a solution of piperidine, and allowing to stand for some time before filtering, but a little $Zn(OH)_2$ is apt to go through the filter, leading to low results. In each case the result of only one experiment is given.

Piperazine cannot be employed, as its dissociation constant is too small (0·0064). The quaternary ammonium bases, *e.g.*, tetramethyl-ammonium hydroxide, which

possess high dissociation constants (0·211), also precipitate magnesium, copper, and zinc salts completely, but the precipitates are so gelatinous that it is almost impossible to wash them completely, and consequently the results obtained are too high.

A. G. L.

Analysis of Cement. **O. H. Klein and S. F. Peckman.** (*Journ. Soc. Chem. Ind.*, 1901, 539.)—The sample for analysis should not be pulverized, but should be exactly like the sample submitted to physical tests. Five grammes are weighed out and gradually introduced into 250 c.c. of not stronger than 10 per cent. hydrochloric acid in such a manner as to avoid any appreciable rise in temperature. The solution should be vigorously stirred at intervals for half an hour. The residue consists of unburnt fuel and ashes. It is separated and weighed. The filtered solution is evaporated to dryness, drenched with concentrated hydrochloric acid, taken up in water, the "soluble silica" filtered off, dried and ignited. The filtrate is made up to 1 litre, and two portions of 100 c.c. each are precipitated with ammonia. The precipitate is filtered off, redissolved in hydrochloric acid, and reprecipitated with ammonia. In the filtrate lime is precipitated as oxalate. Magnesia is next separated. The sulphur may be determined either in the filtrate from the magnesia, or better in the original solution. The only other determination the authors make is the loss on ignition. The "soluble silica," the alumina and iron oxide and the lime, which together constitute the "true cement," should amount to about 90 per cent. The "insoluble in 10 per cent. HCl" in good cements amounts to 3 to 5 per cent., and is considered by the authors to be very harmful. The other impurities they consider to be comparatively harmless.

A. M.

Estimation of Citrate-soluble Phosphoric Acid in Thomas Meal by the Molybdate Method. O. Foerster. (*Chem. Zeit.*, 1901, xxv., 421.)—According to Wagner's directions, the citric acid solution of the phosphoric acid must be placed for ten to fifteen minutes in a water-bath heated to between 80° and 95° C. after adding the ammonium molybdate; and the yellow precipitate must be quickly and entirely soluble in 2 per cent. ammonia. These two regulations are somewhat antagonistic; if the bath is maintained at the temperature quoted, complete solubility is but seldom attained. It is better to put the beaker in a water-bath which has been heated to 80° C., allowing the temperature to fall naturally during the ten or fifteen minutes' immersion, for in that way precipitation of silica is reduced to an insignificant amount. Even a temperature of 60° C. is sufficient to throw down all the phosphoric acid.

The quantity of silica precipitated from the same sample at the same temperature is not always the same, but varies for reasons which have not yet been explained. A thorough investigation of the effect of temperature on the molybdate process would be very welcome.

F. H. L.

ERRATA.

Page 229, 5th line from bottom, *for* "As the figures . . . ." *read* "As the figures given for a commercial malt extract must necessarily be of a purely empirical nature, it was decided . . . ."

Page 230, foot-note, *for* "author's" *read* "authors'."